



**Memorandum**

**To:** Dante Rodriguez, Remedial Project Manager  
Site Cleanup Section 3, SFD-7-3  
USEPA Region 9

**Through:** Joe Eidelberg, Chemist  
Quality Assurance Section, EMD-3-2  
USEPA Region 9

**From:** Kathy O'Brien, Project Manager *Keo*  
ICF, Environmental Services Assistance Team (ESAT) Region 9

ESAT Contract No.: EP-W-13-029  
Technical Direction No.: 10106165

**Date:** July 7, 2017

**Re:** Review of Analytical Data, **Tier 3**

---

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Anaconda Yerington Copper
Site Account No.:	09 GU QB08
Case No.:	46764
SDG No.:	MYA9K9
Laboratory:	Chemtex Environmental Laboratory (CHX)
Analysis:	CLP Metals by ICP-AES and ICP-MS
Samples:	16 Soil Samples
Collection Dates:	April 4, 2017
Reviewer:	Santiago Lee, ESAT

EXES Data Manager has been updated with the results of this review and the validation level revised to S3VEM; the dynamic deliverables were regenerated and are available on the SMO Portal.

If there are any questions, please contact Joe Eidelberg (QA Program/EPA) at (415) 972-3809.

Attachment

cc: Richard Freitas, CLP COR USEPA Region 9  
Raymond Flores, CLP COR USEPA Region 6

CLP PO: ☒ FYI ☐ Action

SAMPLING ISSUES: ☒ Yes ☐ No

10106165-20089/46764/MYA9K9 Rpt



## Data Validation Report – Tier 3

Case No.: 46764  
SDG No.: MYA9K9  
Site: Anaconda Yerington Copper  
Laboratory: Chemtex Environmental Laboratory (CHX)  
Analysis: CLP Metals by ICP-AES and ICP-MS  
Reviewer: Santiago Lee, ESAT  
Date: July 7, 2017

### I. SDG SUMMARY

For Sample Information and Laboratory Quality Control (QC), refer to EXES National Functional Guidelines (NFG) data validation reports *Analytical Sample Listing* and *Inorganic Analytical Sequence*. EXES Data Manager has been updated with the results of this review and the validation level revised to S3VEM; the dynamic deliverables were regenerated and are available on the SMO Portal. The data qualifier definitions, as described in page 6 of the National Functional Guidelines, are attached to this report.

#### Field QC

Field Blanks (FB): None.  
Equipment Blanks (EB): None.  
Background Samples (BG): None.  
Field Duplicates (D1): MYA9L8 and MYA9L9.  
Field Duplicates (D2): MYA9M6 and MYA9M7 (in SDG MYA9M7).

#### CLP PO Action

None.

#### Sampling Issues

1. Samples were received by the laboratory with a temperature of 11.2°C which is above the  $\leq 6^{\circ}\text{C}$  sample preservation criterion (see Additional Comments).
2. Samples were collected on April 4, 2017 and were shipped to the laboratory on April 12, 2017 (see Additional Comments).
3. The SDG Narrative indicates that the samples were received in a box and a temperature blank was not present.
4. The sample log-in sheets indicate that custody seals were not present on the box.

#### Additional Comments

The samples were analyzed for Contract Laboratory Program (CLP) metals. Aluminum, calcium, iron, manganese, potassium, and sodium were analyzed by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). Antimony, arsenic, barium, beryllium, boron, cadmium, chromium, cobalt, copper, lead, magnesium, molybdenum, nickel, selenium, silver, thallium, thorium, uranium, vanadium, and zinc were analyzed by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) under Modification Analysis 2725.0.

As noted in Sampling Issues above, the samples were not adequately maintained at  $\leq 6^{\circ}\text{C}$  as specified in the statement of work (SOW). Technical judgment indicates no adverse effect is expected on metal results; this is substantiated by the recommended preservation criteria for metals in Table 3-2 of EPA publication SW-846, Update V, Revision 5, July 2014 (preservation criteria for metals does not require chilling).

The chain of custody record (COC) indicates that the samples were collected on April 4, 2017 and were shipped to the laboratory on April 12, 2017. The reviewer presumed that the samples

were stored properly from the time of collection to the time of shipping. The laboratory documentation indicates that they were received at a temperature of 11.2°C.

For samples MYA9M4 and MYA9M5, results for beryllium and boron were incorrectly reported from the undiluted analysis instead of the 2-fold dilution. The laboratory submitted revised Form 1A upon request, on June 30, 2017; the corrected results are reported in the EXES Data Manager.

For the ICP-MS analysis, true values and recoveries for molybdenum were incorrectly reported on Form 4 (ICP Interference Check Sample). The laboratory submitted revised Form 4 upon request, on June 30, 2017. Recoveries for molybdenum are within the QC limit.

The preparation records for some solutions were missing in the data package. The laboratory submitted the missing pages upon request, on June 30, 2017. All standards and spiking solutions were analyzed before the expiration date.

The samples listed below did not meet the internal standard QC limit of 60-125% relative intensity (RI) in the initial analysis. The samples were diluted and reanalyzed as required by the SOW. Results for beryllium and boron in samples MYA9M0, MYA9M4, and MYA9M5 are reported from the 2-fold dilution because the RIs are within the QC limit.

Sample	Internal Standard	% Relative Intensity from Undiluted	% Relative Intensity from 2-Fold Dilution
MYA9M0	Lithium-6	59	62
MYA9M4	Lithium-6	58	60
MYA9M5	Lithium-6	58	60

This report was prepared in accordance with the following documents:

- ☐ USEPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration), ISM02.4, October 2016;
- ☐ ICP-MS Analysis Plus Boron, Molybdenum, Thorium, and Uranium, Modified Analysis 2725.0, February 8, 2017; and
- ☐ USEPA National Functional Guidelines for Inorganic Superfund Data Review, January 2017.

For technical definitions, refer to *Exhibit G (Glossary of Terms)*, USEPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration), ISM02.4.

## II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

	<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1.	Data Completeness	Yes	
2.	Preservation and Holding Times	Yes	
3.	ICP-MS Tune Analysis	Yes	
4.	Calibration	Yes	
	a. Initial	Yes	
	b. Initial and Continuing Calibration Verification	Yes	
5.	Laboratory Blanks	Yes	B
6.	Field/Equipment Blanks	N/A	
7.	ICP Interference Check Sample (ICS)	Yes	
8.	Laboratory Control Sample (LCS)	Yes	
9.	Duplicate Sample Analysis	Yes	
10.	Spike Sample Analysis	No	C
11.	ICP Serial Dilution	No	D
12.	ICP-MS Internal Standards	Yes	
13.	Analyte Quantitation	Yes	A, F

	<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
14.	Field Duplicate Sample Analysis	No	E
15.	Overall Assessment of Data	Yes	

N/A = Not Applicable.

### III. VALIDITY AND COMMENTS

- A. Results above the method detection limit (MDL) but below the contract required quantitation limit (CRQL) are estimated and flagged "J." Results are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in analytical precision near the quantitation limit.
- B. The following results are qualified as non-detected (U) due to low level initial calibration blank (ICB), continuing calibration blank (CCB), and preparation blank (PB) contamination.
- ☐ Antimony in all samples except MYA9K9 and MYA9L6.
  - ☐ Boron in samples MYA9L3, MYA9L4, MYA9M3, and MYA9M5.
  - ☐ Molybdenum in samples MYA9L5 through MYA9L7, MYA9M0, MYA9M3, and MYA9M5.
  - ☐ Selenium in preparation blank PBSE48.

Analyte amounts greater than the MDL but less than the CRQL were reported in calibration blanks and preparation blank at the concentrations presented below.

Analyte	Blank	Concentration, µg/L
Antimony	ICB	0.32
Boron	ICB/CCBS76/CCBS77/CCBS81	4.5/2.4/2.1/1.2
Molybdenum	PBSE48	0.20 mg/kg
Selenium	CCBS76/CCBS80/CCBS81	0.81/0.86/0.80

Sample results that are greater than or equal to the MDL but less than or equal to the CRQL are reported as non-detected (U) at the respective CRQL.

- C. The following results are estimated and flagged "J+" because a matrix spike recovery is outside the method QC limit.
- ☐ Thorium in all field samples.

Matrix spike recovery for thorium in QC sample MYA9L3S does not meet the 75-125% criterion for accuracy as presented below.

Analyte	% Recovery
Thorium	135

The detected results for thorium are considered quantitatively uncertain and may be biased high.

The following post-digestion spike recovery was reported for thorium in QC sample MYA9L3A. The post-digestion spike recovery does not reflect the entire sample preparation and analysis; the impact on reported results cannot be determined. Qualification is based on the matrix spike recovery only.

Analyte	Post-Digestion Spike, % Recovery
Thorium	35

Since both the post- and pre-digestion spikes do not meet the QC criteria, the unacceptable pre-digestion spike recovery may indicate poor laboratory technique or matrix effects which may interfere with the analysis.

- D. The following results are estimated and flagged “J” because a serial dilution result is outside the method QC limit.

☐ Manganese in all field samples.

Percent difference for serial dilution analysis of MYA9L3L does not meet the 10% difference criterion for the analyte presented below.

Analyte	% Difference
Manganese	11

Results for manganese are considered quantitatively uncertain. Chemical and physical interferences may exist due to sample matrix effects. Since the result for diluted sample is lower than the original, the reported results may be biased high.

- E. Results for the following field duplicate pair do not meet the absolute difference criterion as presented below.

Analyte	MYA9M6 (D2), mg/kg	MYA9M7 (D2), mg/kg	Difference, mg/kg	Limit, mg/kg
Thorium	5.1	3.3	1.8	1.0

Results for the following field duplicate pair do not meet the relative percent difference (RPD) criterion for precision as presented below.

Analyte	MYA9M6 (D2), mg/kg	MYA9M7 (D2), mg/kg	RPD	QC Limit
Uranium	2.2	1.7	26%	20%

This uncertainty should be evaluated in the context of project data quality objectives to determine data usability.

- F. Samples MYA9L2, MYA9L5, MYA9L8, MYA9L9, MYA9M2, and MYA9M6 were reanalyzed at 2.0-fold dilutions due to concentrations of manganese exceeding the calibration range. Results for manganese in these samples are reported from the diluted analyses.

## DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "National Functional Guidelines for Inorganic Superfund Data Review," January 2017 (Table 1, page 6).

- U      The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J      The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+     The result is an estimated quantity, but the result may be biased high.
- J-     The result is an estimated quantity, but the result may be biased low.
- UJ     The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
- R      The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.